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Effect of Load Rate on Ultimate Tensile Strength of Ceramic Matrix Composites at Elevated Temperatures

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EFFECT OF LOAD RATE ON ULTIMATE TENSILE STRENGTH OF CERAMIC MATRIX COMPOSITES AT ELEVATED TEMPERATURES

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ABSTRACT

Strength of three continuous fiber-reinforced ceramic composites, including SiC/CAS-II, SiC/MAS–5 and SiC/SiC, was determined as a function of test rate in air at 1100 to 1200 °C. All three composite materials exhibited a strong dependency of strength on test rate, similar to the behavior observed in many advanced monolithic ceramics at elevated temperatures. The application of the preloading technique as well as the prediction of life from one loading configuration (constant stress-rate) to another (constant stress loading) suggested that the overall macroscopic failure mechanism of the composites would be the one governed by a power-law type of damage evolution/accumulation, analogous to slow crack growth commonly observed in advanced monolithic ceramics. It was further found that constant stress-rate testing could be used as an alternative to life prediction test methodology even for composite materials, at least for short range of lifetimes and when ultimate strength is used as the failure criterion.

INTRODUCTION

The successful development and design of continuous fiber-reinforced ceramic composites (CFCCs) are dependent on a thorough understanding of basic properties such as fracture and delayed failure (slow crack growth, fatigue, or damage accumulation) behavior. Particularly, accurate evaluation of delayed failure behavior under specified loading/environment conditions is a prerequisite to ensue accurate life prediction of structural components.

This paper describes the effect of load rate on elevated-temperature ultimate tensile strength of three different Nicalon™ fiber-reinforced ceramic composites including SiC_f/calcium-aluminosilicate (CAS), SiC_f/magnesium-aluminosilicate (MAS) and SiC_f/silicon-carbide (SiC) ceramic composites. For each composite material, strength was determined in air as a function of test rate at elevated temperature of 1100 °C (SiC/CAS and SiC/MAS) or 1200 °C (SiC/SiC). This type of testing, when used for monolithic ceramics, is called "constant stress-rate" or "dynamic fatigue" testing [1 to 3]. The loading rate dependency of strength was analyzed with the power-law (damage) propagation, conventionally utilized for monolithic ceramics and glass. Preloading tests were conducted to better understand the governing failure mechanism(s) of the materials. Finally, the result of elevated-temperature constant stress ("static fatigue" or "stress rupture") testing was obtained for each material and compared with that of constant stress rate testing. This was done to further verify the overall failure mechanism of the material and to establish constant stress-rate testing as a means of life prediction test methodology for CFCCs. It should be noted that few studies on these subjects have been done for continuous fiber-reinforced ceramic composites [4], particularly at elevated temperatures.

EXPERIMENTAL PROCEDURE

All the matrices of the three test composites were reinforced by ceramic-grade Nicalon™ fibers with a fiber volume fraction of about 0.39. The nominal fiber diameters ranged from 10 to 15 μm. The three composite materials included Nicalon™ unidirectionally (1D) fiber-reinforced calcium aluminosilicate (designated SiC/CAS-II), Nicalon™ crossply (2D) magnesium aluminosilicate (designated SiC/MAS–5), and Nicalon™ plain-woven (2D) silicon carbide composites. SiC/CAS-II and SiC/MAS–5 were fabricated by Corning, Inc., through hot-pressing followed by ceraming of the composites by a thermal process. The designation "–5" in SiC/MAS–5 indicates that the matrix was doped with 5 vol% fraction of borosilicate glass. The silicon carbide matrix in the SiC/SiC composites was fabricated by the DuPont Company through chemical vapor infiltration (CVI) into the fiber perform. SiC/CAS-II and SiC/MAS–5 laminates were 18 and 16 plies thick, respectively, with a nominal thickness of about 3 mm. The plain-woven laminates of the SiC/SiC composite were supplied 12 plies (normally 3.5 mm) thick. More detailed information regarding the test composite materials can be found elsewhere [5]. The SiC/CAS-II material has been used in a previous, preliminary study on test rate-effect on tensile strength [6]. The dogboned tensile test specimens measuring 152.4 mm (length) × 12.7 mm (width) were machined from the composite laminates, with the gage section of about 30 mm long, 10 mm wide and 3.0 to 3.5 mm thick (as-furnished). The design of the dogboned tensile test specimen was the result of previous finite element analysis [7].

Monotonic tensile testing was conducted in air at 1100 °C for both SiC/CAS-II and SiC/MAS–5 and at 1200 °C for SiC/SiC, using a servohydraulic test frame (Model 8501, Instron, Canton, MA). A total of three to four different loading rates (in load control), corresponding to stress rates ranging within 50 to 0.005 MPa/s, were employed with typically 3 test specimens tested at each loading rate. Detailed experimental procedure on tensile testing and related induction-heating equipment can be found elsewhere [5]. Preloading or accelerated testing technique, applied primarily to monolithic ceramics and glass [8], was also conducted at test temperatures using 0.5 MPa/s (for SiC/CAS-II) or 0.005 MPa/s (for SiC/MAS–5 and SiC/SiC) in an attempt to better understand the governing failure mechanism of the materials. Predetermined preloads, corresponding to about 80 to 90 percent of the failure strength determined at 0.5 or 0.005 MPa/s with zero preload (regular testing), were applied quickly to the test specimens prior to testing and their corresponding strengths were measured. Typically two to three test specimens were used in preload testing. Tensile testing was performed in accordance with ASTM Test Standard, ASTM C 1359 [9].

Constant stress ("static fatigue" or "stress rupture") tensile testing was also performed in air for the three composite materials using the same test specimens, test fixture, test frame and same test temperatures that were used for monotonic tensile testing. The limited availability of test materials confined the testing to four to nine test specimens, depending on material. Four different static loads were applied to test specimens and their corresponding times to failure were determined.

RESULTS

Constant Stress-Rate Testing

The results of monotonic tensile strength testing with different test rates are presented in Fig. 1, where *ultimate strength* was plotted as a function of *applied stress rate* for each composite material using log-log scales. Each solid line in the figure indicates a best-fit regression line based on the log (*ultimate strength*) versus log (*applied stress rate*) relation. The decrease in ultimate strength with decreasing stress rate, which represents a susceptibility to damage accumulation or delayed failure, was significant for all the composite materials. The strength degradation was about 51, 31 and 62 percent, respectively, for SiC/CAS-II, SiC/MAS–5 and SiC/SiC

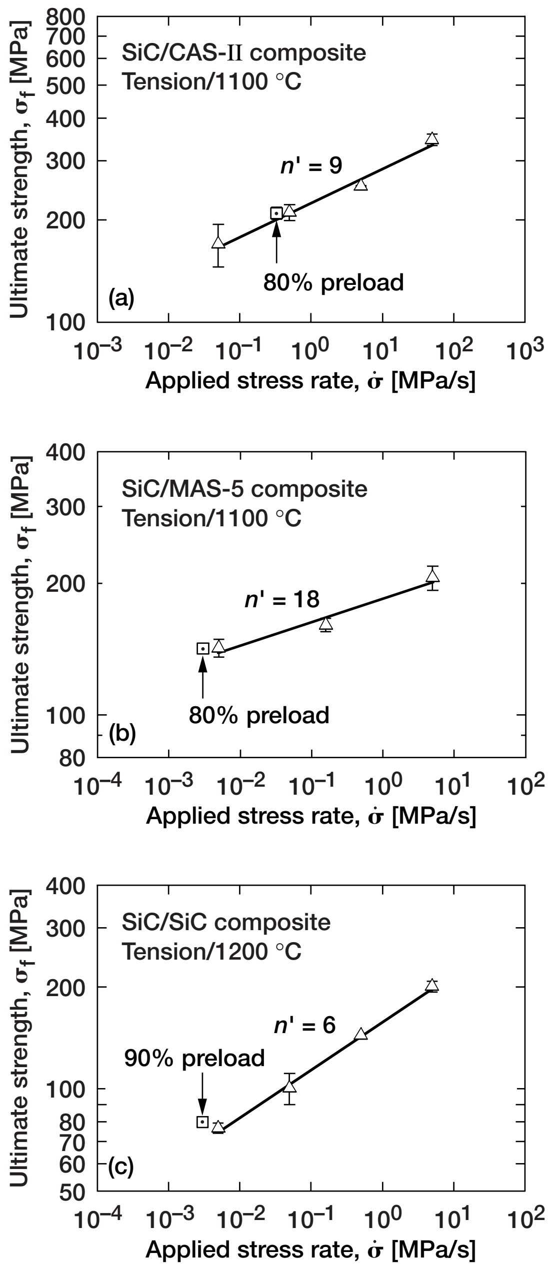

Figure 1.—Results of ultimate tensile strength as a function of stress rate for (a) SiC/CAS-II, (b) SiC/MAS-5, and (c) SiC/SiC composites at elevated temperatures in air. The solid lines represent the best-fit regression lines. Error bar indicates ±1.0 standard deviation. The results of preloading tests are also included.

when stress rate decreased from the highest to the lowest. Fracture patterns for the SiC/CAS-II composite showed some fiber pullout with jagged faceted matrix cracking often propagating along the test-specimen length, as shown in Fig. 2. For a given stress rate, however, the difference in strength between different fracture patterns was not obvious. No appreciable difference in the mode of failure was observed for SiC/MAS–5 and SiC/SiC, where most specimens tested at either high or low stress rate exhibited relatively flat fracture surfaces (see Fig. 2), possibly called *brittle fracture*.

Preload Testing

The results of preloading tests are also shown in Fig. 1, where the ultimate strength with 80 to 90 percent preloads is compared with that in regular testing with zero preload. The difference in strength between two preloads (0 and 80 to 90 percent) was negligibly small for each material: 211 MPa (for 0 percent preload) and 209 MPa (for a 85 percent preload) for SiC/CAS-II; 142 MPa (0 and 80 percent preload) for SiC/MAS–5; 77 MPa (0 percent) and 80 MPa (90 percent) for SiC/SiC. Hence, the maximum strength difference, exhibited by SiC/SiC, amounts to only about 4 percent. This indicates that any significant damage that would control ultimate strength of the material did not occur before the applied loads up to 80 to 90 percent of fracture load. Conversely, the damage to control final failure would have occurred when applied load or test time was greater than 80 to 90 percent of fracture load or total test time. The theory explaining results of preload testing will be described in the discussion section.

Constant Stress ("Stress Rupture") Testing

A summary of results of constant stress or stress rupture testing at elevated temperatures is presented in Fig. 3, where *time to failure* was plotted against *applied stress* for each composite material using log-log scales. A significant decrease in time to failure with increasing applied stress, which represents a susceptibility to damage accumulation or delayed failure, was evident for all the composite materials tested. Each solid line in Fig. 3 indicates a prediction made based on the constant stress-rate data (Fig. 1), which will be discussed in a later section. The mode of fracture in constant stress testing was very similar to that in constant stress-rate testing. Brittle failure was exemplified for SiC/MAS–5 (2D) and SiC/SiC (2D), while somewhat jagged matrix cracking was observed for SiC/CAS-II (1D).

DISCUSSION

The strength dependency on test rate exhibited by the three composite materials (Fig. 1) is very similar to that observed in advanced monolithic ceramics at ambient or elevated temperatures. The strength degradation with decreasing stress rate has been known to be due to slow crack growth (delayed failure or fatigue) of an initial crack, typically governed by the following empirical power-law relation [1 to 3]

$$v = \alpha\left(K_I / K_{IC}\right)^n \tag{1}$$

where v, K_I and K_{IC} are crack velocity, mode I stress intensity factor and fracture toughness, respectively. α and n are called slow crack growth (SCG) parameters. Based on this power-law relation, the strength (σ_f) can be derived as a function of applied stress rate ($\dot{\sigma}$) [1 to 3].

$$\sigma_f = D[\dot{\sigma}]^{1/n+1} \tag{2}$$

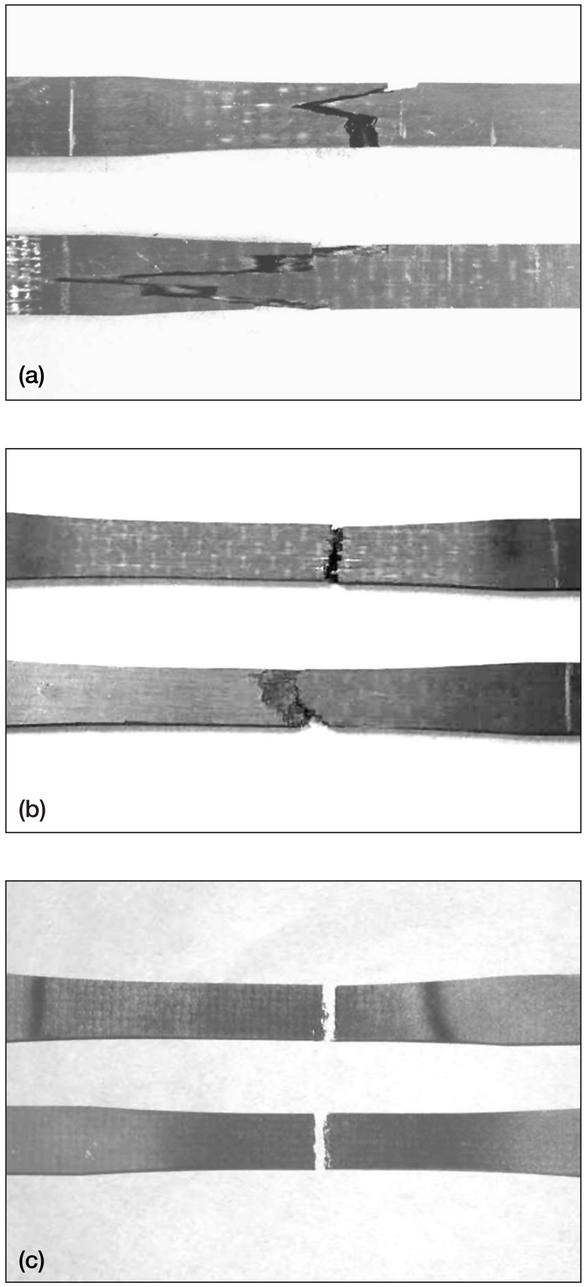

Figure 2.—Fracture patterns for (a) SiC/CAS-II, (b) SiC/MAS-5, and (c) SiC/SiC composites subjected to elevated-temperature tensile testing. The upper and lower pictures for a given composite material indicate the specimens tested at the lowest and the highest load rates, respectively.

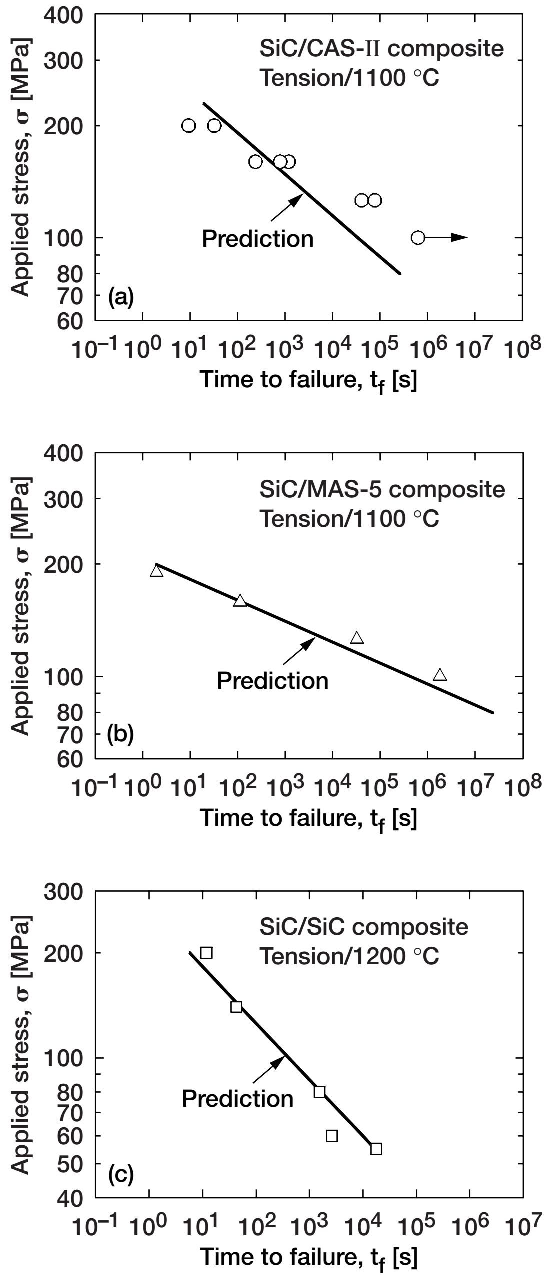

Figure 3.—Results of constant stress ("static fatigue" or "stress rupture") testing for (a) SiC/CAS-II, (b) SiC/MAS-5, and (c) SiC/SiC composites at elevated temperatures in air. The solid lines represent the predictions made based on the results of constant stress-rate testing (Figure 1).

where D is another SCG parameter associated with inert strength, n and crack geometry. Equation (2) can be expressed in a more convenient form by taking logarithms of both sides

$$\log \sigma_f = \frac{1}{n+1} \log \dot{\sigma} + \log D \tag{3}$$

Constant stress-rate ("dynamic fatigue") testing based on Eqs. (2) or (3) has been established as ASTM Test Methods (C1368 [2] and C1465 [3]) to determine SCG parameters of advanced monolithic ceramics at ambient and elevated temperatures. It has been recommended to use units of MPa for σ_f and MPa/s for $\dot{\sigma}$ [2 to 3]. As can be seen in Fig. 1, the data fit to Eq. (3) is very reasonable with the coefficients of correlation (r_{coef}) all greater than 0.980, indicating that the damage evolution/accumulation or delayed failure of the composite materials would be adequately described by the power-law type relation, Eq. (1). Assuming this, the *apparent* parameters n' and D' for the composites were determined using a linear regression analysis based on Eq. (3) with the data in Fig. 1. Values of n' = 9.0 and D' = 226, n' = 18 and D' = 185, and n' = 6 and D' = 158 were obtained for SiC/CAS-II, SiC/MAS–5 and SiC/SiC, respectively (The prime was used here for composite materials to distinguish them from monolithic ceramic counterparts.). It is noteworthy that the value of n', a measure of susceptibility to damage, was very low for both SiC/CAS-II and SiC/SiC, but intermediate for SiC/MAS–5. Typical monolithic silicon nitrides and silicon carbides at high temperatures at ≥1200 °C exhibit $n \geq 20$. Hence, compared with monolithic ceramics, the SiC/CAS-II and SiC/SiC composites exhibited a significantly higher susceptibility to damage evolution/accumulation.

The preload or accelerated test technique has been developed for monolithic ceramics in order to save test time in constant stress-rate testing [8]. Based on *the power-law SCG* relation of Eq. (1) with some mathematical manipulation, strength of a test specimen under a preload (α_p) was derived as a function of preloading factor [8,2, and 3];

$$\sigma_{fp} = \sigma_f \left(1 + \alpha_p^{n+1}\right)^{\frac{1}{n+1}} \tag{4}$$

where σ_{fp} is strength with a preload and α_p ($0 \leq \alpha_p \leq 1$) is a preloading factor (or percentage of preload) in which a preload stress (applied to the test specimen) is normalized with respect to the strength with zero preload. Equation (4) indicates that strength with a preload is sensitive to the magnitude of preload particularly at lower n and higher α_p values. A theoretical prediction of ultimate strength as a function of preload, based on Eq. (4) with estimated values of n' from Fig. 1, is shown in Fig. 4. The prediction (solid lines) is in excellent agreement with the experimental data for all the three composite materials tested, as seen in the figure. This result obtained for the composite materials is also analogous to that observed in advanced monolithic ceramics and glass [8]. Damage, mainly SCG, of monolithic ceramics occurs substantially close to 90 percent of total failure time because of their higher n (≥20) value [8]. The applicability of the preloading analysis for the composite materials strongly suggests that major damage evolution/accumulation process would be the one governed by the power-law relation (Eq. (1)) and that the damage would have occurred after a long incubation time, at least after 80 percent of total test time.

For the case that a single delayed failure mechanism (SCG) is predominant, a life prediction (for monolithic ceramics) from one loading configuration to another can be made analytically or numerically, depending on the complexity of loading configurations concerned. A prediction

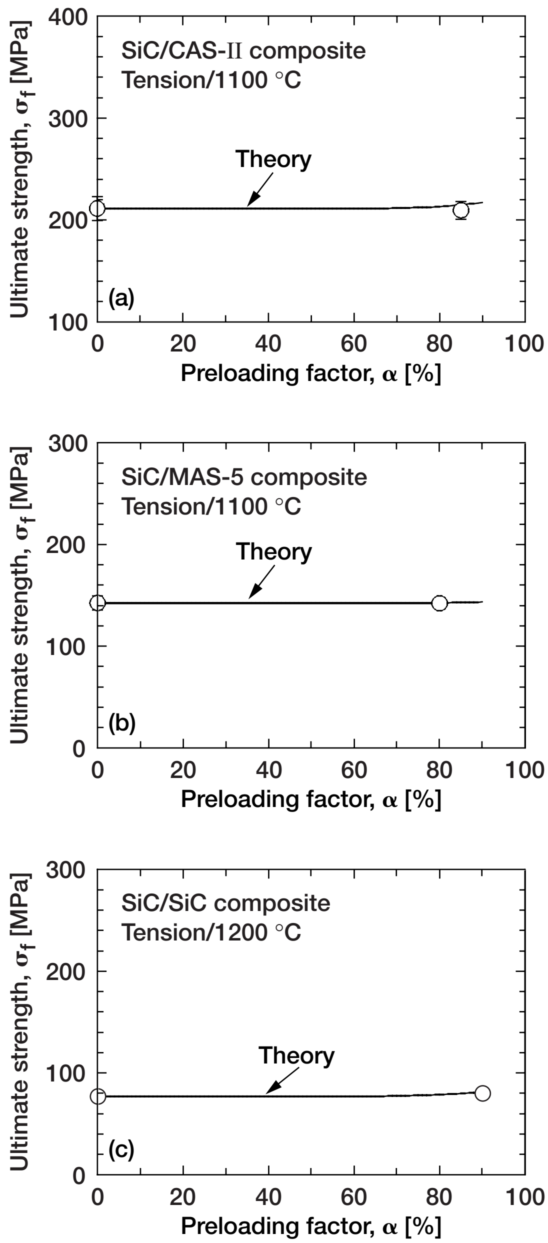

Figure 4.—Result of preloading tests (ultimate strength as a function of preloading) for (a) SiC/CAS-II, (b) SiC/MAS-5, and (c) SiC/SiC composites at elevated temperatures in air. A theoretical line based on Equation (4) [8] is included for comparison for each composite material.

of life in constant stress loading can be made based on Eq. (1) using constant stress-rate testing data as follows:

$$t_f = \left[\frac{D^{n+1}}{n+1}\right]\sigma^{-n} \tag{5}$$

where t_f and σ are time to failure and applied stress, respectively. Use of Eq. (5) together with n' and D' determined in constant stress-rate testing allows one to predict life in constant stress loading. The results thus predicted are presented in Fig. 3 as solid lines. Except for some discrepancy in the SiC/CAS-II composite, the overall prediction is in reasonable agreement with experimental data, at least for short periods of life. This indicates that the governing failure mechanism of SiC/MAS–5 and SiC/SiC was identical in both constant stress rate testing and constant stress testing. Since the prediction (Eq. (5)) was made based on the power-law relation, it is certain that the distinct failure mechanism of the two composite materials would be governed by the power-law type of damage evolution/accumulation (or SCG or delayed failure).

The strength dependency on test rate, the applicability of preloading technique and the reasonable life prediction from one loading configuration (constant stress-rate testing) to another (constant stress or stress rupture testing) all support that the damage evolution/accumulation of the composite materials tested was controlled by a process very similar to the power-law type of SCG of monolithic ceramics, and that the failure mechanism was almost independent of loading configuration either in monotonically increased or in constant loading. This indicates that the constant stress-rate testing, commonly utilized in determining life prediction parameters of monolithic ceramics, could be applicable even to composite materials. The merit of constant stress rate testing is enormous in terms of simplicity and test economy (short test time) over other stress rupture or cyclic fatigue testing, especially for short lifetimes. A continuing effort to establish a database in constant stress rate testing at elevated temperatures is in progress using more ceramic matrix composites. A more detailed study regarding microscopic failure mechanisms [4, 10 to 13] associated with matrix/fiber interaction, matrix cracking and its effect on slow crack growth, and delayed failure of sustaining fibers near fracture, etc. is needed. Finally, the results of this work also suggest that care must be exercised when characterizing elevated-temperature strength of composite materials. This is due to that fact that elevated-temperature strength has a relative meaning if a material exhibits rate dependency: the strength simply depends on which test rate one chooses (Fig. 1). Therefore, at least two test rates (high and low) are recommended to better characterize the high-temperature strength behavior of a composite material.

CONCLUSIONS

Elevated-temperature strength of three continuous fiber-reinforced ceramic composites, including SiC/CAS-II, SiC/MAS–5 and SiC/SiC, exhibited a strong dependency on test rate, similar to the behavior observed in many advanced monolithic ceramics at elevated temperatures. The applicability of the preloading technique as well as the predictability of life from one loading configuration (constant stress-rate) to another (constant stress loading) suggested that the distinct, overall failure mechanism of the composite materials would be a process primarily governed by a power-law type of damage evolution/accumulation, analogous to the mechanism observed in monolithic counterparts. It was further found that constant stress-rate testing could be utilized as a means of life prediction test methodology even for composites when short lifetimes are expected.

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13. ABSTRACT *(Maximum 200 words)*

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